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(21) International Application Number: PCT/US92/04502 (22) International Filing Date: 29 May 1992 (29.05.92) (30) Priority data: 707,339 29 May 1991 (29.05.91) US (71) Applicant: UTAH STATE UNIVERSITY FOUNDATION [US/US]; 1780 North Research Park Way, Suite 108, North Logan, UT 84321 (US). (72) Inventors: BROWN, Rodney, J. ; 615 Circle Place, Providence, UT 84331 (US). SAVELLO, Paul ; 85 North 400 East, Hyde Park, UT 84318 (US). McMAHON, Donald, J. ; 719 East 400 South, Smithfield, UT 84335 (US).		(74) Agents: ROBBINS, Beatrice, N. et al.; Venable, Baetjer, Howard & Civiletti, 1201 New York Avenue, N.W., Suite 1000, Washington, DC 20005 (US). (81) Designated States: AT (European patent), AU, BE (European patent), BR, CA, CH (European patent), CS, DE (European patent), DK (European patent), ES (European patent), FI, FR (European patent), GB (European patent), GR (European patent), HU, IT (European patent), JP, KR, LU (European patent), MC (European patent), NL (European patent), NO, PL, RO, RU, SE (European patent). Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: CONCENTRATED STERILE MILK PRODUCT (57) Abstract A process for preparing aseptically packaged concentrated milk is disclosed. To avoid the use of a stabilizer and allow the storage of concentrated milk in packages without refrigeration and without the formation of age gelatin, milk products are concentrated by reverse osmosis, the concentrated milk products are then sterilized by an ultra high sterilization process, the milk is optionally homogenized and the sterilized product is then aseptically packaged.		

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CONCENTRATED STERILE MILK PRODUCT

Field of The Invention

The invention relates to an improved method for concentrating fresh milk products and aseptically packaging the concentrates. The improved packaged products are free of stabilizers and keep at grocery shelf temperatures for several months without age gelation. The concentrates can be reconstituted with water to make fresh-tasting milk that has the mouth-feel of fresh milk and is consumer-acceptable as "fresh milk". Specifically, the method comprises concentrating fresh milk by reverse osmosis, sterilizing under UHT conditions, homogenizing and aseptically packaging.

BACKGROUND OF THE INVENTION

Milk products such as whole milk, low fat milk and skim milk are generally preferred by the consumer in their fresh form. However, fresh milk products have drawbacks because the fresh products do not keep for more than a few days and must be kept under virtually constant refrigeration during those few days.

In order to reduce shipping costs, reduce storage costs and to achieve longer storage time, it has been known to concentrate milk by evaporation and to aseptically package the concentrate so that it can be kept fit for human consumption for longer periods of time.

The Ranjith patent, USP 4,921,717 relates to concentrating milk by evaporation and subsequently packaging the concentrate aseptically.

Although it has been known in the art to concentrate milk products using reverse osmosis, the prior art has not dealt with a process comprising UHT processing, aseptic packaging, and long term storage, wherein the packaged product does not require the addition of stabilizer.

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SUMMARY OF THE INVENTION

The present invention relates to a packaged concentrated sterile milk product and to a new process for production of packaged concentrated sterile milk products.

Concentrated sterile milk of the invention is a dairy product which will have a worldwide market. It has proved particularly valued for its long keeping properties without refrigeration and the fact that it will retain its fresh qualities for these long periods. This is especially important in many countries without their own dairy industries, where the product of the invention will be diluted for use in place of fresh milk, or used as is for coffee cream.

The production process for manufacturing and packaging the inventive concentrated sterile milk involves the separation of water from the milk supply by pumping the milk supply stream through reverse osmosis (RO) filters until the solids content is as desired; passing the milk concentrate through a UHT (Ultra High Temperature) sterilization process, and aseptically packaging the concentrated sterile milk into containers sized to fit the market segment as needed. Packaging may be in packages as small as 1 ounce to 1 gallon for home or other use, or in up to 300 gallon bulk packages for export or interplant shipment.

The superior color and flavor of the concentrated sterile milk product of the invention has now been recognized. The product also has the advantage of not using stabilizers or other additives to prevent gelation during long term storage.

There are an increasing number of countries which insist that milk and milk products should be free from stabilizers and there is a general trend towards the avoidance of such materials wherever possible.

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This invention provides a process which removes only water from the milk and therefore leaves the balance of all other elements as in the original milk. It is believed that this balance is responsible for natural prevention of gelation during storage.

It has been found that the disadvantages of prior processes can be avoided by the use of the inventive process in which milk is R.O. concentrated.

Accordingly, the present invention provides a process for producing a concentrated sterile milk product comprising the following steps of

- (a) concentrating milk or milk product,
- (b) sterilizing the concentrate in an appropriate manner, and
- (c) aseptically packaging under the sterile conditions.

After opening the aseptic package, water may be added to reconstitute the product, returning it to a normal solids content for use as fresh milk.

DETAILED DESCRIPTION

The process of the present invention is a process for making aseptically packaged concentrated milk from fresh milk feedstock comprising (a) concentrating the fresh milk feedstock by reverse osmosis; (b) sterilizing the concentrate, and (c) aseptically packaging the concentrate wherein only water is removed from the feedstock, and wherein spontaneous formation of a gel in said concentrate during unrefrigerated storage is avoided without the use of stabilizers or other additives to prevent age gelation.

The process of the present invention may be used for concentrating, sterilizing, and packaging any milk or fluid milk product, hereinafter referred to as "feedstock". The feedstock of the invention may be raw or pasteurized. The feedstock can be other milk

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products such as cream and buttermilk. Whole milk is generally a milk with a milk fat content of at least 3.0%. Skim milk is defined in most states as having a maximum milk fat content of 0.1-0.5%. Low fat milk has a fat content falling between the fat content of whole milk and skim milk. Generally, low fat milk has a fat content in the 1% - 2% range.

In the example of the invention, the feedstock is cow's milk although it is contemplated that the milk of any mammal might be used, for instance goat's milk.

The concentrate may be sterilized by any sterilization method as long as the balance of all elements of a feedstock except water is not adversely disturbed. Preferably the concentrate is sterilized by a UHT continuous flow process wherein the product is passed over one or more heat exchangers or is sterilized by a steam heated process. Heating rates and holding times may be selected as convenient for the equipment in use.

The process can be built to operate as a continuous flow of the feedstock through the process or a batch process with the feedstock being held for a period between steps of the process. In one embodiment, the process may be interrupted after step (a) and the concentrate can be transported to a different site before being subjected to steps (b) and (c). In this case, it may be convenient to conduct step (a) at the farm or at a processing plant, with steps (b) and (c) being performed at a distance away. The product may then be transported and/or stored for a period of time before being reconstituted for use as fresh milk. Alternately, the concentrate of step (a) can be mixed with similarly processed concentrate.

Preferably the process is conducted in a continuous manner in which case step (a) suitably involves a reduction in volume to half or less than half the volume

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of the original feedstock. Most preferably, step (a) involves concentrating feedstock to one third of its original volume, with a solids content of approximately 34% for a whole milk feedstock and 24% for a skim milk feedstock.

The process of the invention offers several advantages including improved energy efficiency in the concentration/sterilization plant as well as in the transportation of the products. In addition, the need for refrigerated storage in stores and at home is eliminated.

In one embodiment of the invention, it is contemplated that flavoring or other suitable additives that enhance taste or mouth feel might be added to the concentrate before it is aseptically packaged. One particularly popular flavor that might be added is chocolate flavor.

The concentrate may be packaged in large containers. It is contemplated that containers with a capacity of up to 300 gallons would be suitable for use in the wholesale market. In a further embodiment, the large packages of concentrate for the wholesale market might be aseptically repackaged into smaller sized containers for the retail market.

The process of the invention is new and surprisingly useful because it has been discovered that the aseptically packaged concentrate of the invention does not spontaneously form a gel at normal storage temperatures for periods of 6 months or more. Normal storage temperatures can be defined as temperatures as low as refrigerator temperatures up to about 80°F. It has been determined that higher storage temperature are permissible. If the storage temperature is no higher than about 75°F, the packaged product will not gel spontaneously and will retain its flavor for at least one year.

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The equipment that was used to sterilize the concentrate included means for homogenizing the milk and therefore the milk was homogenized. However, homogenization can be an optional step. It is contemplated that in an alternate embodiment the feedstock could be homogenized before it is concentrated.

EXPERIMENTAL WORK LEADING TO THE INVENTION

As a first step in the research that led to the invention, a non-cellulosic R.O. membrane, identified as Type ZF 99, was obtained from the Damrow Company of Fond du Lac, Wisconsin. The membrane was made by Patterson Candy International, Ltd.

In the example reported below, 100 gallons of fresh milk was R.O. processed using this membrane. The milk was heated to about 130°F (54°C) before being R.O. processed. However, it is not necessary to heat the milk before it is concentrated. Heating the milk allowed the process to be carried out a little faster.

After the milk was concentrated, it was sterilized under UHT conditions, homogenized and aseptically packaged in four-ounce clear plastic sterile containers. The containers were cylindrical and rigid.

When 100 ml of milk was added to the container, the container was filled up to a level of 10 cm. In order to test the keeping qualities of the stabilizer-free concentrate as compared with concentrate containing stabilizer, four sets of packaged milk were prepared. One (1) ml of an additive was combined with the 100 ml of milk in each package before the package was capped. A first set of packages (set 1) illustrate the invention. The additive of set 1 was one (1) ml of sterile pure water. The packages in the other three sets each contained one (1) ml of a salt solution (a

stabilizer). In set 2, set 3 and set 4, the salt solutions used were: one (1) ml 10% sodium citrate; one (1) ml 10% disodium phosphate; and one (1) ml 10% sodium hexametaphosphate respectively. The samples were stored at room temperature ($75^{\circ}\text{F} \pm 5^{\circ}\text{F}$).

Periodically, samples were observed in their undisturbed state and the observations were recorded. Periodically, three samples from each set were warmed in an incubator ($30^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$), opened and an amount of the enzyme rennet was added to each sample. The samples were then allowed to incubate for at least 30 minutes before the samples were observed and were pH tested. The enzyme used was New Zealand rennet with an activity 185RU/ml. Each of the three samples was treated with 0.93, 1.85, and 3.70 RU/ml of sample respectively. As it turned out, the results observed and the pH values were about the same regardless of which of the three concentrations of enzyme was added to the sample.

Making Milk Concentrate

Raw milk with a fat content of 3.37%, a total solids content of 12.45%, and a pH of 6.50 was heated to about 130°F and concentrated by reverse osmosis with a non-cellulosic membrane (Type ZF99) obtained from Paterson Candy International Limited, having an operative area of 18.85 sq. ft., using an initial permeate rate of 1100 ml per min at a pressure of 500 psi. The milk was concentrated to total solids content of 25.0%.

Sterilizing and Homogenizing Concentrate

Under UHT Conditions

Concentrate was preheated to 177°F (80.5°C); UHT processed at 284°F (120°C) for about 4 seconds and homogenized at a temperature of about $135\text{--}136^{\circ}\text{F}$ ($57^{\circ}\text{C} - 58^{\circ}\text{C}$) using pressures of 2000 psi (first stage) and 500

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psi (second stage). Sterilization and homogenization was done using an Alfa Laval Sterilab System.

Aseptic Packaging of Samples

Concentrate was packaged at about 126°F (52°C) manually in sterile plastic containers using an aseptic laminar flow hood. The heated milk was added to the 100 ml mark on each of the plastic containers.

In addition to the milk, one (1) ml water was added to each package illustrating the invention, Set 1. Similarly one (1) ml of a solution of each of: 10% sodium citrate 10% disodium phosphate; and 10% sodium hexameta-phosphate was added to each package of set 2, set 3 and set 4. Samples of concentrate were observed at room temperature without disturbing the closed container. The results of these observations are set forth in Table 1. Some samples of concentrate were enzyme treated, incubated for at least 30 minutes and then observed and tested. The results are set forth in Table 2.

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OBSERVATIONS RELATED TO STABILITY OF
UNDISTURBED ROOM TEMPERATURE PACKAGED
WHOLE MILK CONCENTRATE OVER TIME

Set 1 - No Stabilizer Added

<u>Storage Time</u> <u>In Weeks</u>	<u>Observations</u>
12	no observable change
15	slight cream ring and no
sediment	
18	no change, a small 1mm layer
	of white on bottom
21	cream ring, about 3mm sediment
	on bottom
24	about 2mm sediment on bottom
27	about 2mm sediment on bottom
33	2mm sediment on bottom
39	2mm sediment on bottom
46	4mm sediment on bottom

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Set 2 - Sodium Citrate Added

<u>Storage Time</u> <u>In Weeks</u>	<u>Observations</u>
15	slight cream ring, no sediment
18	one mm layers of white on bottom
21	cream ring, 3mm layer of sediment
24	do
27	do
33	do
39	do
46	3mm layer of sediment; signs of gelation on edge of container

Set 3 - Disodium Phosphate Added

<u>Storage Time</u> <u>In Weeks</u>	<u>Observations</u>
15	slight cream ring, no sediment
18	visible separation into layers and 4mm sediment
21	gelled clear whey/liquid, some separation of particles
24	do
27	curd seems a lot softer. Not as much separation between the whey and curd as before.
33	gelled curd and whey seem like they are coming together again
39	gelled curd and whey seem to be softening
46	gelled.

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Set 4 - Sodium hexameta phosphate added

<u>Storage Time</u> <u>In Weeks</u>	<u>Observations</u>
15	slight cream ring and no sediment
18	do
21	do
24	do
27	do
33	do
39	do
46	do

Note: The presence of a cream ring or sediment in the concentrate did not have a permanent effect on the concentrate. If a sample only showed signs of a cream ring and/or sediment, it was possible to shake the concentrate in order to eliminate the cream ring and/or sediment. After shaking, the concentrate returned to a stable system.

TABLE 2

OBSERVATIONS AND TEST RESULTS RELATED TO THE
STABILITY
OF INCUBATED ENZYME - TREATED WHOLE MILK CONCENTRATE

Set 1 - no stabilizer added

<u>Storage Time</u> <u>in Weeks</u>	<u>pH</u>	<u>Observations</u>
0	6.48	coagulation
3	6.38	slight cream ring
6	6.41	no change
9	6.32	no change
36	6.33	3mm sediment on bottom
50	6.24	no change

Set 2 - Sodium citrate added

<u>Storage Time</u> <u>in Weeks</u>	<u>pH</u>	<u>Observations</u>
0	6.55	no coagulation
3	6.43	slight cream ring
6	6.47	do
9	6.40	no change
36	6.40	3mm sediment at bottom
50	—	6mm sediment, gelled in middle of container

Set 3 - Disodium phosphate added

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<u>Storage Time</u> <u>in Weeks</u>	<u>pH</u>	<u>Observations</u>
0	6.58	no coagulation
3	6.46	slight cream ring
6	6.51	do (except one gelled and had ph 4.75)
9	6.46	no change
36	6.43	gelled, seem to be softening
50	—	gelled, seems very soft

Set 4 - Sodium hexametaphosphate added

<u>Storage Time</u> <u>In Weeks</u>	<u>pH</u>	<u>Observations</u>
0	6.45	no coagulation
3	6.29	slight cream ring
6	6.33	do
9	6.30	no change
36	6.25	no change, no sediment
50	6.17	smells rancid

Note: The presence of a cream ring or sediment in the concentrate did not have a permanent effect on the concentrate. If a sample only showed signs of a cream ring and/or sediment, it was possible to shake the concentrate in order to eliminate the cream ring and/or sediment. After shaking, the concentrate returned to a stable system.

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Conclusions To Be Drawn From
Data In Tables 1 and 2

The data shows that the aseptically packaged stabilizer-free concentrate of the invention can be kept at usual room storage temperatures (not under refrigeration) for more than 6 months without showing any signs of age gelation.

When a stabilizer is added to the concentrate, and the stabilizer is sodium citrate or disodium phosphate, the stabilizer does not prevent age gelation and in fact appears to cause age gelation. Age gelation is evident in the concentrate as early as the 21st week of storage.

When the stabilizer is sodium hexametaphosphate, the stabilizer does not appear to have any effect one way or the other on age gelation.

The data indicates that the stabilizer-free milk concentrate of the invention will keep on the grocer's shelf or under like conditions for more than 6 months without showing age gelation. The concentrate of the invention has a stability which makes it possible to make a reconstituted milk product from the concentrated that is equivalent to fresh milk in gel stability.

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We Claim:

1. A process for making aseptically packaged concentrated milk from fresh milk feedstock comprising:
 - a) concentrating the fresh milk feedstock by removing water from said feedstock by reverse osmosis;
 - b) sterilizing the concentrate of step (a);and
 - c) aseptically packaging said concentrate,wherein spontaneous formation of a gel in said concentrate during unrefrigerated storage is avoided without use of stabilizers or other additives to prevent gelation.
2. The process of claim 1 wherein the feedstock is selected from a group consisting of cream, whole milk, low fat milk, and skimmed milk.
3. The process of claim 1 wherein said sterilizing is UHT sterilizing.
4. The process of claim 3 wherein the package size of said packages is not less than about one ounce and not more than about 300 gallons.
5. The process of claim 3 wherein said concentrate is free of stabilizer or any other additive used to prevent gel formation.
6. The process of claim 5 wherein the packaged concentrate comprises flavoring or other additive.
7. The process of claim 6 wherein said flavoring is chocolate.

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8. The process of claim 1 wherein the volume of said concentrate is no less than about one-third the volume of said feedstock.

10. A process for making aseptically packaged concentrated whole milk from a volume of fresh whole milk comprising:

a) feeding the volume of whole milk to a reverse osmosis (RO) system thereby to remove water and form a volume of milk concentrate;

b) sterilizing the concentrate under UHT conditions;

c) optionally homogenizing; and

d) aseptically packaging the concentrate into packages.

11. The process of claim 10 wherein the size of said packages is not less than about one ounce and not more than about 300 gallons.

12. The process of claim 10 wherein said feedstock is pasteurized feedstock.

13. The process of claim 10 wherein said volume of fresh whole milk is reduced to a volume of concentrate not less than about one-half of the volume of said fresh whole milk and wherein said concentrate has a solids content of up to about 25% by weight.

14. The process of claim 13 wherein said volume of fresh whole milk is reduced to a volume of concentrate not less than about one-third of the volume of said fresh whole milk and wherein said concentrate has a solids content of up to about 34% by weight.

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15. The process of claim 10 wherein said concentrate is free of stabilizer or any other additive used to prevent gel formation.

16. A process for making packages of aseptically packaged concentrated skim milk from a volume of fresh skim milk comprising:

a) feeding the volume of skim milk to a reverse osmosis (RO) system thereby to remove water and form a volume of skim milk concentrate;

b) sterilizing the concentrate under UHT conditions;

c) optionally homogenizing; and

d) aseptically packaging.

17. The process of claim 16 wherein the size of said packages is not less than about one ounce and not more than about 300 gallons.

18. The process of claim 17 wherein said feedstock is pasteurized feedstock.

19. The process of claim 16 wherein the volume of said fresh skim milk feedstock is reduced to a concentrate with a volume no less than about one-third the volume of said fresh skim milk feedstock.

20. The process of claim 19 wherein said concentrate has a solids content of up to about 24% by weight.

21. A process for making packages of aseptically packaged concentrated low fat milk from a volume of fresh low fat milk comprising:

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a) feeding the volume of low fat milk to a reverse osmosis (RO) system to thereby remove water and form a volume of low fat milk concentrate;

b) sterilizing under UHT conditions;

c) optimally homogenizing; and

d) aseptically packaging.

22. The process of claim 21 wherein the size of said packages is not less than about one ounce and not more than about 300 gallons.

23. The process of claim 22 wherein said feedstock is pasteurized feedstock.

24. The process of claim 22 wherein the volume of the fresh low fat milk feedstock is reduced to a concentrate with a volume no less than about one-third the volume of said fresh low fat milk.

25. A process for preparing aseptically packaged concentrated milk from a volume of raw milk feedstock comprising the steps of:

a) pasteurizing the volume of raw milk feedstock;

b) feeding the pasteurized feedstock to a reverse osmosis (RO) system and thereby removing water from the feedstock and forming a volume of concentrate wherein said volume is no less than about one-half the volume of the raw milk feedstock;

c) sterilizing the concentrate under UHT conditions;

d) homogenizing; and

e) aseptically packaging said concentrate in packages.

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26. The process of claim 25 wherein said package size is about 100 ml.

27. The process of claim 26 wherein the volume of said concentrate is about half the volume of the raw milk feedstock.

28. The aseptically packaged concentrate made by the process of claim 1.

29. The reconstituted milk product made by the addition of water to the concentrate of claim 1 wherein said reconstituted milk has essentially the same balance of the elements as the fresh milk feedstock.

30. The aseptically packaged concentrate made by the process of claim 10.

31. The reconstituted whole milk made by the addition of water to the concentrate of claim 30 wherein said reconstituted milk has essentially the same balance of elements as fresh whole milk.

32. The aseptically packaged concentrate of claim 20.

33. The reconstituted skim milk made by the addition of water to the concentrate of claim 32 wherein the reconstituted milk has essentially the same balance of elements as fresh skim milk.

34. The aseptically packaged concentrate of claim 21.

35. The reconstituted low fat milk made by the addition of water to the concentrate of claim 34 wherein

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said reconstituted milk has essentially the same balance of elements as fresh low fat milk.

36. A process for making aseptically packaged concentrated milk from two or more amounts of fresh milk feedstock comprising:

(a) concentrating each of said two or more amounts of fresh milk feedstock separately by reverse osmosis;

(b) combining the concentrates formed by step (a);

(c) sterilizing; and

(d) aseptically packaging.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) : A23C 1/00; A23C 3/02

US CL : 426/399, 400, 422, 491, 522, 587

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 426/492

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US, A, 4,921,717 (Ranjith) 01 May 1990, See entire document.	26-33
Y		1-8,10-36
Y	Dairy Foods, February 1990, pages 71-73, See entire document.	1-8,10-36
Y	Paterson Candy International, January 1983, See entire document.	1-8,10-36
Y	Dairy Industries, Vol. 36, No. 9, September 1971, pages 507-509, See entire document.	1-8,10-36
Y	Chemical Engineering Progress, December 1968, pages 31-43, See page 41, column 1.	1-8,10-36
Y	US, A, 3,552,574 (Lowe et al) 05 January 1971, See column 1, line 16-29 and column 5, line 9-33.	1-8,10-36

☐

Further documents are listed in the continuation of Box C.

☐

See patent family annex.

*

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